

What is claimed is:

1. A non-oxide powder of which at least about 40% by mass is comprised of a plurality of tightly agglomerated compositions, each of said tightly agglomerated compositions comprising a plurality of primary particles tightly agglomerated together, wherein said 5 primary particles have an average size in a range of about 1 nanometer to about 500 nanometers.

2. A non-oxide powder of claim 1 wherein for each tightly agglomerated composition, at least about 60% of its constituent primary particles are within  $2.25dN^{1/3}$  nanometers of at least about 60% of its other constituent primary particles, wherein N represents 5 the number of primary particles in the tightly agglomerated composition, and wherein d represents the average size of the primary particles in the tightly agglomerated composition.

3. The non-oxide powder of claim 2 wherein said primary particles are selected from the group consisting of metals, intermetallics, metal matrix composites, ceramics, and non-metals.

4. The non-oxide powder of claim 3 wherein, for each tightly agglomerated composition, at least about 60% of its constituent primary particles are within  $2.25dN^{1/3}$  nanometers of all its other primary particles.

5. The non-oxide powder of claim 3 wherein, for each tightly agglomerated composition, at least about 50% of its constituent primary particles have sizes not deviating by more than a factor of 2 from the average size of its constituent primary particles, and at 5 least about 90% of its constituent primary particles have sizes not deviating by more than a factor of 4 from the average size of its primary particles.

6. The non-oxide powder of claim 3 wherein, for each tightly agglomerated composition, at least about 80% of its primary particles are within  $2.25dN^{1/3}$  nanometers of at least about 80% of its other primary particles.

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7. The non-oxide powder of claim 3 wherein each of said tightly agglomerated compositions are comprised of at least 20 of said primary particles agglomerated together.

8. The non-oxide powder of claim 7 wherein, for each tightly agglomerated composition, at least about 60% of its primary particles are not closer than  $dN^{1/3}$  nanometers of at least about 60% of its other primary particles.

9. The non-oxide powder of claim 3 wherein at least about 75% by mass of said powder is comprised of said tightly agglomerated compositions.

10. The non-oxide powder of claim 3 wherein at least one of said tightly agglomerated compositions further comprises an secondary material within which the primary particles of said at least one agglomerated composition are encapsulated.

11. The non-oxide powder of claim 10 wherein said secondary material is selected from the group consisting of salts and polymers.

12. The non-oxide powder of claim 11 wherein each of said tightly agglomerated compositions further comprises a secondary material within which its primary particles are encapsulated.

13. The non-oxide powder of claim 11 wherein said powder has an encapsulation efficiency in the range of about 10% to about 99% by mass.

14. The non-oxide powder of claim 13 wherein said powder has an encapsulation efficiency in the range of about 70% to about 99% by mass.

15. The non-oxide powder of claim 3 wherein said primary particles are selected from the group consisting of metals.

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16. The non-oxide powder of claim 18 wherein said primary particles are selected from the group consisting of valve metals.

17. The non-oxide powder of claim 16 wherein said primary particles are tantalum.

18. The non-oxide powder of claim 17 wherein each of said tightly agglomerated compositions has a specific surface area in the range of about 1 m<sup>2</sup>/g to about 70 m<sup>2</sup>/g.

19. The non-oxide powder of claim 16 wherein said primary particles are niobium.

20. The non-oxide powder of claim 15 wherein said primary particles are selected from the group consisting of refractory metals.

21. The non-oxide powder of claim 3 wherein said primary particles are selected from the group consisting of carbon and boron. 40

22. The non-oxide powder of claim 3 wherein said primary particles are selected from the group consisting of ceramics. 20

23. The non-oxide powder of claim 3 wherein said primary particles are selected from the group consisting of intermetallics. 50

24. The non-oxide powder of claim 3 wherein said primary particles are selected from the group consisting of metal matrix composites. 40

25. The non-oxide powder of claim 3 wherein said powder has a primary particle purity based on metal contaminants of less than about 10,000 parts per million by mass.

26. The non-oxide powder of claim 25 wherein said powder has a primary particle purity based on metal contaminants of less than about 1000 parts per million by mass.

27. The non-oxide powder of claim 26 wherein said powder has a primary particle purity based on metal contaminants of less than about 50 parts per million by mass.

28. The non-oxide powder of claim 1 wherein said primary particles have an average size in the range of about 1 nanometer to about 100 nanometers.

29. A method of producing agglomerated submicron particles, said method comprising:

nucleating a plurality of primary particles in an environment of a desired temperature and comprised of a vaporous secondary material;

5 creating a mixture of a plurality of encapsulated primary particles and a plurality of unencapsulated primary particles by condensing said secondary material upon a fraction of said primary particles once at least two of said primary particles have grown to a size in the range of about 1 nanometer to about 500 nanometers, wherein said fraction is about 1/5 or less;

10 controlling the temperature of said environment while said encapsulated primary particles scavenge said unencapsulated primary particles such that (1) a plurality of encapsulated primary particle clusters are created, each encapsulated primary particle cluster comprising a plurality of co-encapsulated primary particles, and (2) said co-encapsulated primary particles in said encapsulated primary particle clusters sinter.

15 30. The method of claim 29 wherein said primary particles are selected from the group consisting of metals, intermetallics, ceramics, metal matrix composites, and non-metals.

31. The method of claim 30 wherein said secondary material is selected from the group consisting of salts and polymers.

32. The method of claim 31 wherein said nucleating step comprises reacting a vaporous reactive metal and a metallic halide or halides

in a reaction zone having a gas environment, said reaction producing said primary particles and said secondary material.

33. The method of claim 32 wherein said reacting step comprises introducing a continuous flow of a said vaporous reactive metal and a continuous flow of said vaporous metallic halide into said reaction zone for flame synthesis.

34. The method of claim 33 wherein said introducing step comprises controlling the rate of flow, temperature, and/or composition of one or both of said vaporous reactive metal and said vaporous metallic halide such that said co-encapsulated primary particles in said

5 encapsulated primary particle clusters sinter.

35. The method of claim 29 wherein said fraction is about 1/5 or less.

36. A method of producing agglomerated submicron particles, said method comprising:

5 creating a vaporous mixture of a desired pressure and temperature and comprising at least a vaporous primary material and a vaporous secondary material; and

10 controlling the pressure and temperature of said vaporous mixture to (1) grow discrete primary particles of said primary material to a size within the range of approximately 1 nanometer to approximately 500 nanometers, (2) condense said secondary material upon less than all of said primary particles once at least two of said primary particles has grown to said size, thereby creating a mixture of a plurality of encapsulated primary particles and a plurality of unencapsulated primary particles, (3) allow said encapsulated primary particles to scavenge said unencapsulated primary particles, thereby creating a plurality of primary particle clusters, each primary particle cluster comprising an encapsulated primary particle and at least one scavenged primary particle, and (4) sinter said primary particles in said primary particle clusters.

37. The method of claim 36 wherein said primary material is selected from the group consisting of metals, intermetallics, metal matrix composites, ceramics, and non-metals.

38. The method of claim 37 wherein said secondary material is selected from the group consisting of salts and polymers.

39. A method of producing agglomerated submicron particles, said method comprising:

5 mixing a gaseous reactive metal with a metallic halide in a closed reaction zone having a gas environment, thereby creating a reaction, said reaction producing a plurality of nucleating primary particles and a condensable secondary material; and

10 creating a plurality of primary particle agglomerations by (1) controlling the temperature and pressure of said environment to be greater than condensation level for said secondary material and (2) reducing the temperature of at least a portion of said environment to thereby force the condensation of said secondary material upon a fraction of said primary particles once at least two of said primary particles have grown to a size in the range of about 1 nanometer to about 500 nanometers, wherein said fraction is about 1/5 or less, 15 and wherein said temperature is controlled such that at least some of said primary particles upon which said secondary material has condensed sinter with other primary particles to thereby form an agglomeration of primary particles.

40. The method of claim 39 wherein said creating step includes controlling the temperature and pressure of said environment such that the time between collisions of said primary particles in said environment is less than a coalescence time for said primary 5 particles.

41. The method of claim 39 wherein said fraction is about 1/5 or less.

42. The method of claim 39 further comprising controlling the duration of the creating step such that said primary particles are

partially encapsulated for an encapsulation efficiency in the range of about 10% to about 99% by mass.

43. The method of claim 39 further comprising controlling the duration of the scavenging mode such that all of said primary particles are encapsulated.

44. The method of claim 39 wherein said primary particles are tantalum particles, and wherein said creating step includes controlling the temperature of said environment to be within a range of about 600°C to about 1500°C.

45. A method of producing agglomerated submicron particles, said method comprising:

synthesizing discrete primary particles in the presence of a vaporous secondary material, all within a gas environment; and

5 thermodynamically controlling the condensation of said secondary material to (1) encapsulate less than all of said primary particles once said primary particles grow to a size within the range of approximately 1 nanometer to approximately 500 nanometers, thereby producing a mixture of a plurality encapsulated primary particles and a plurality of unencapsulated primary particles, and (2) allow the encapsulated primary particles and unencapsulated primary particles to collide with each other and sinter to form encapsulated agglomerations of primary particles.

10 46. The method of claim 45 wherein said primary particles are selected from the group consisting of metals, intermetallics, ceramics, metal matrix composites, and non-metals.

47. The method of claim 46 wherein said secondary material is selected from the group consisting of salts and polymers.

48. The method of claim 47 wherein the synthesizing step includes introducing a continuous flow of a vaporized halide and a continuous flow of a vaporized reactive metal into a burner for flame synthesis.

49. The method of claim 47 wherein said thermodynamically controlling step includes controlling the sintering of said primary particles that have collided with each other such that the time between collisions of said primary particles is less than a 5 coalescence time for said primary particles.

50. The method of claim 47 wherein said primary particles are tantalum particles, and wherein said thermodynamic controlling step includes controlling the temperature of said environment to be within a range of about 600°C to about 1500°C.

51. A powder comprising:

a plurality of primary particles, each primary particle having a size in the range of about 1 nanometer to about 500 nanometers; and

5 a secondary material within which at least some of said primary particles are encapsulated.

52. The powder of claim 51 wherein a plurality of said primary particles are agglomerated together.

53. The powder of claim 52 wherein between about 10% and about 100% of said primary particles are encapsulated in said secondary material.

54. The powder of claim 53 wherein said primary particles are selected from the group consisting of metals, intermetallics, metal matrix composites, ceramics, and non-metals.

55. The powder of claim 54 wherein said secondary material is selected from the group consisting of salts and polymers.

56. The powder of claim 55 wherein said primary particles are selected from the group consisting of metals.

57. The powder of claim 56 wherein said primary particles are selected from the group consisting of valve metals.

58. The powder of claim 57 wherein said primary particles are tantalum.

59. The powder of claim 58 wherein said agglomeration of tantalum particles has a specific surface area in the range of about 1 m<sup>2</sup>/g to about 70 m<sup>2</sup>/g.

60. The powder of claim 56 wherein said primary particles are selected from the group consisting of refractory metals.

61. The powder of claim 55 wherein said primary particles are selected from the group consisting of carbon and boron. *No*

62. The powder of claim 55 wherein said primary particles are selected from the group consisting of ceramics. *XO*

63. The powder of claim 55 wherein said primary particles are selected from the group consisting of intermetallics. *XO*

64. The powder of claim 55 wherein said primary particles are selected from the group consisting of metal matrix composites. *XO*

65. The powder of claim 55 wherein said secondary material is NaCl.

66. The powder of claim 55 wherein said secondary material is removable.

67. A capacitor-grade powder of which at least about 40% by mass is comprised of a plurality of tight agglomerations of primary particles, each tight agglomeration comprising at least 20 primary particles, said primary particles having an average size in the range of about 1 nanometer to about 200 nanometers and being selected from the group consisting of valve metals, wherein for each of said tight agglomerations, at least about 60% of its constituent primary particles are within  $2.25dN^{1/3}$  nanometers of at least about 60% of its other constituent primary particles, wherein N represents

10 the number of primary particles in the same tight agglomeration, and wherein d represents the average size of said primary particles.

68. The capacitor-grade powder of claim 67 wherein said powder has a primary particle purity based on metal contaminants of less than about 1000 parts per million by mass.

69. The capacitor-grade powder of claim 67 wherein at least one of said tight agglomerations further comprises a secondary material within which the primary particles of that tight agglomeration are encapsulated, said secondary material being selected from the group 5 consisting of salts and polymers.

70. The capacitor-grade powder of claim 67 wherein said powder has an encapsulation efficiency in the range of about 70% to about 100% by mass.

71. A method of producing metal matrix composites, said method comprising:

synthesizing discrete primary particles in the presence of a vaporous secondary material, all within a gas environment; 5 thermodynamically controlling the condensation of said secondary material to thereby encapsulate a fraction of said primary particles once at least two of said primary particles have grown to a size within the range of approximately 1 nanometer to approximately 500 nanometers, thereby creating a mixture of a plurality of encapsulated primary particles and a plurality of 10 unencapsulated primary particles, wherein said fraction is about 1/5 or less;

15 exposing said unencapsulated primary particles to an environment reactive with said unencapsulated primary particles, thereby causing said unencapsulated primary particles to react with said reactive gas environment to form a plurality of dispersion particles;

exposing said encapsulated primary particles and said dispersion particles to a non-reactive environment;

20 removing said secondary material from said encapsulated primary particles in said non-reactive environment; and consolidating said primary particles with said dispersion particles to thereby form a metal matrix composite.

72. The method of claim 71 wherein said primary particles are selected from the group consisting of aluminum, copper, magnesium, titanium, zinc, and alloys and combinations thereof.

73. The method of claim 72 wherein said secondary material is selected from the group consisting of salts and polymers.

74. The method of claim 73 wherein said reactive environment is comprised of gas selected from the group consisting of (1) oxygen, (2) air, (3) nitrogen, (4) ammonia, (5) boron trichloride, (6) carbon tetrachloride, (7) some combination of gases (1)-(6), and (8) some mixture comprised of any of said gases (1)-(7) mixed with an inert gas.

75. The method of claim 74 wherein said step of exposing unencapsulated primary particles to said reactive environment further comprises exposing said encapsulated primary particles to said reactive environment, wherein said reactive environment is not reactive with said secondary material.

76. A metal matrix composite comprised of:

at least about 50% by mass of a plurality of encapsulated primary particle agglomerations, each encapsulated primary particle agglomeration comprising a plurality of agglomerated primary particles and a secondary material within which its primary particles are encapsulated; and

the balance comprising a plurality of dispersion particle agglomerations, each dispersion particle agglomeration comprising a plurality of agglomerated primary particles combined with a dispersion material; and

wherein each of said primary particles have a size within the range of about 1 nanometer to about 500 nanometers.

77. The metal matrix composite of claim 76 wherein said dispersion material is selected from the group consisting of oxides, nitrides, carbides, borides, and hydrides.

78. The metal matrix composite of claim 77 wherein at least about 75% by mass of said metal matrix composite is comprised of said encapsulated primary particle agglomerations.

79. The metal matrix composite of claim 78 wherein at least about 95% by mass of said metal matrix composite is comprised of said encapsulated primary particle agglomerations.